भारतीय मानक
Indian Standard

IS 11959 : 2023

कठोर धातुओं में सूक्ष्म संरचना के मेटालोग्राफिक का निर्धारण — पद्धति

(पहला पुनरीक्षण)

Metallographic Determination of Microstructure in HardMetals — Method

(First Revision)

ICS 77.160

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भारतीय मानक ब्यूरो
BUREAU OF INDIAN STANDARDS
मानक भवन, 9 बहादुर शाह ज़फर मार्ग, नई दिल्ली - 110002
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI - 110002
www.bis.gov.in www.standardsbis.in

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Powder Metallurgical Materials and Products Sectional Committee, MTD 25

FOREWORD

This Indian Standard (First Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Powder Metallurgical Materials and Products Sectional Committee had been approved by the Metallurgical Engineering Division Council.

This standard was first published in 1987. This revision has been brought out to bring the standard in the latest style and format of the Indian Standards.

In addition, the following changes have been made:

a) Reference clause has been added;
b) In **4**, an apparatus (**4.2**) scanning electron microscope has been included;
c) In **4**, an apparatus (**4.3**) electron back scatter diffraction (EBSD) has been included;
d) In Fig. 2 has been updated; and
e) In Fig. 3 and Fig. 4 are added.

In preparation of this standard, assistance has been derived from the following standard:

ISO 4499-1 : 2020	Hardmetals — Metallographic determination of microstructure

The composition of the Committee responsible for the formulation of this standard is given in Annex A.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 2022 ‘Rules for rounding off numerical values (*second revision*)’. The number of significant places retained in the rounded off value should be same as that of the specified value in this standard.

Indian Standard

METALLOGRAPHIC DETERMINATION OF MICROSTRUCTURE IN HARD METALS — METHOD

(First Revision)

1 SCOPE

This standard specifies the methods of metallographic determination of the microstructure of hard metals.

2 REFERENCE

The standard given below contain provisions which through reference in this text, constitute provision of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent edition of this standards:

IS No.	*Title*
IS 11520 : 1985	Method for metallographic sample preparation of hardmetals

3 DEFINITION OF SYMBOL

Sl No. (1)	*Symbol* (2)	*Definition* (3)
i)	α - phase	Tungsten carbide (WC)
ii)	β - phase	Binder phase (for example, Co, Ni, Fe)
iii)	γ - phase	Carbide of a cubic lattice (for example: TiC, TaC, NbC) that may contain other carbides (for example: WC) in solid solution
iv)	η - phase	Multiple carbides of tungsten and at least one metal of the binder phase

4 APPARATUS

4.1 Optical Microscope — capable of magnifications up to 1 500 X

4.2 Scanning Electron Microscope — for magnification over 1 500 X

4.3 Electron Back Scatter Diffraction (EBSD)

4.4 Equipment — for the metallographic preparation of test specimen

5 SPECIMEN PREPARATION

5.1 Test specimen shall be selected and prepared for metallographic examination as per the procedure given in IS 11520.

5.2 The surface to be examined shall be free from grinding and polishing mark. Care shall be taken to avoid tearing out of particles, which may lead to a wrong evaluation of microstructure.

6 PROCEDURE

6.1 Examine the microstructure by gradual development of the phases by etching. Examples of suitable etching techniques are given in Table 1. Care shall be taken to ensure that the true microstructure is revealed.

6.2 Determine the presence of η - type phases by lightly etching the section with technique (I) (*see* Table 1). Phases of η - type are coloured orange to brown. γ - phase may etch lightly, while the other phases remain unetched. Etching by technique (I) does not prevent subsequent etching by techniques (II) or (III). Examine the entire section at low magnification and, if necessary, at magnification up to 1 500 X. Note and record the existence of η - type phases and their distribution.

6.3 Determine the presence of γ - phases by etching with technique (II) (*see* Table 1). This phase appears light yellowish brown and has a typically rounded shape (*see* Fig. 1). Examine the etched section and note and record the existence of γ - phase. Estimate and record its size according to Fig. 1 as γ - fine, γ - medium, or γ - coarse.

6.4 Determine the presence of α - phase by etching the surface with technique (III), or in case γ - phase is present, with technique (II) (*see* Table 1). The α - phase appears grey and often has an angular shape. Examine the etched section and note and record the presence of an α - phase. Estimate and record its size according to Fig. 2, Fig. 3 and Fig. 4 as α - fine, α - medium, or α - coarse. Nano, ultrafine, submicron or extra coarse (*see* Fig. 2, Fig. 3 *and* Fig. 4).

6.5 Identify the β - phase after etching the surface by technique (II) or technique (III) (*see* Table 1). This phase remains white.

7 TEST REPORT

The test report shall include the following information:

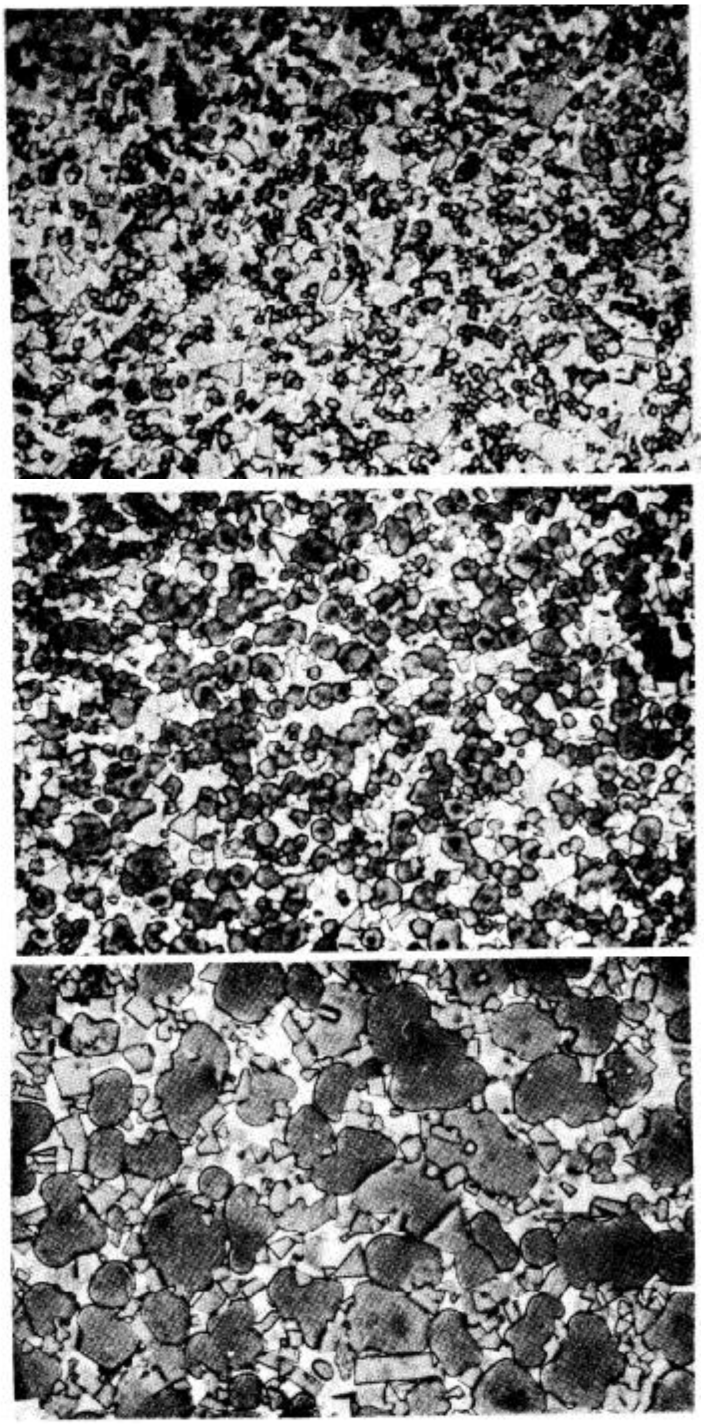
a) Reference to this standard;
b) Complete identification of the test sample;
c) The results obtained; and
d) Details of any occurrence which may have affected the result.

Table 1 Etching Technique

(*Clauses* 6.1, 6.2, 6.3, 6.4 *and* 6.5)

Sl No. (1)	**Etching Technique** (2)	**Composition of Etchants** (3)	**Conditions of Etching** (4)	**Application of Etching** (5)
i)	I	Solution A — Freshly prepared solution of equal quantities of 10 percent to 20 percent (m/m) aqueous solutions of potassium hexacyanoferrate (III) (potassium ferricyanide) and potassium or sodium hydroxide.	Etch in solution A at approximately 20 °C for 2 s to 20 s. Flush the text piece section with water immediately, with surface carefully with acetone or alcohol without wiping.	Identification of η - phases
ii)	II	Solution A — *See* technique (I) Solution B — A mixure of equal volumes of concentrated hydrochloric acid and water.	Etch at approximately 20 °C in Solution A for 3 min to 5 min. Then wash in water and etch in Solution B for approximately 10 s. Next wash in water, then in alcohol and dry the test-piece section. Finally, etch in Solution A for approximately 20 s.	Identification of γ - phase
iii)	III	Solution A — *See* technique (I)	Etch in Solution A at approximately 20 °C for 3 min to 6 min.	Identification of α - phase

NOTE — The separate solutions of potassium hexacyanoferrate (III) and potassium or sodium hydroxide may be stored for a long time, but must be freshly mixed each day, when used.

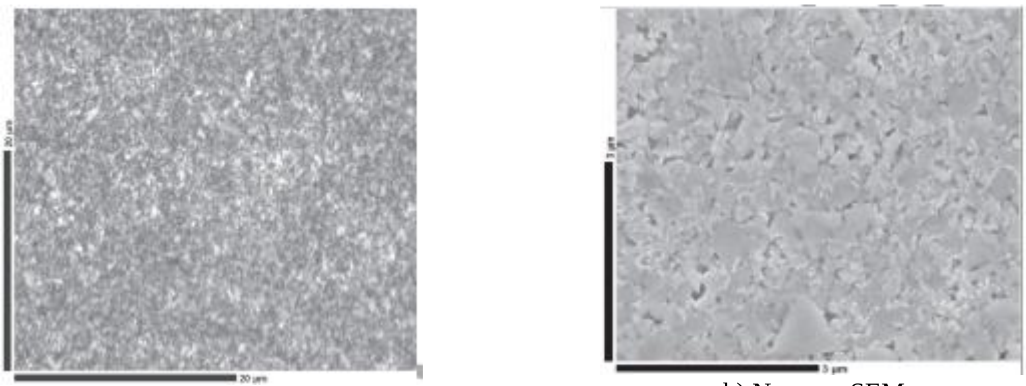


γ - fine

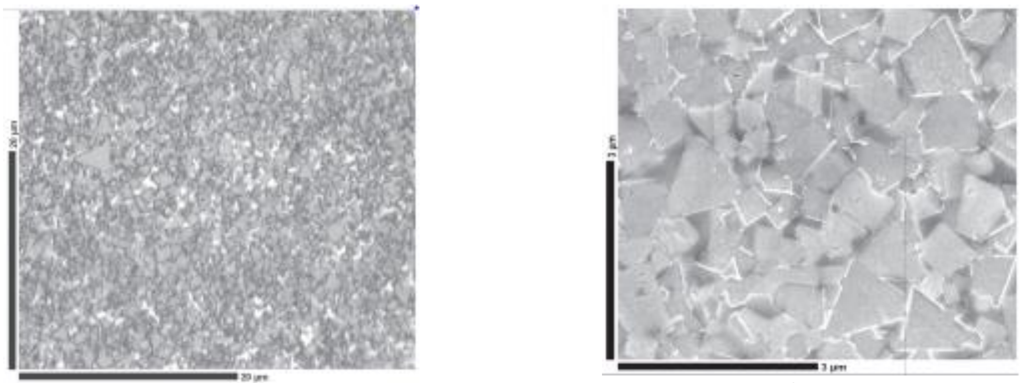
γ - medium

γ - coarse

FIG. 1 γ - PHASE (AT 1 500 X)

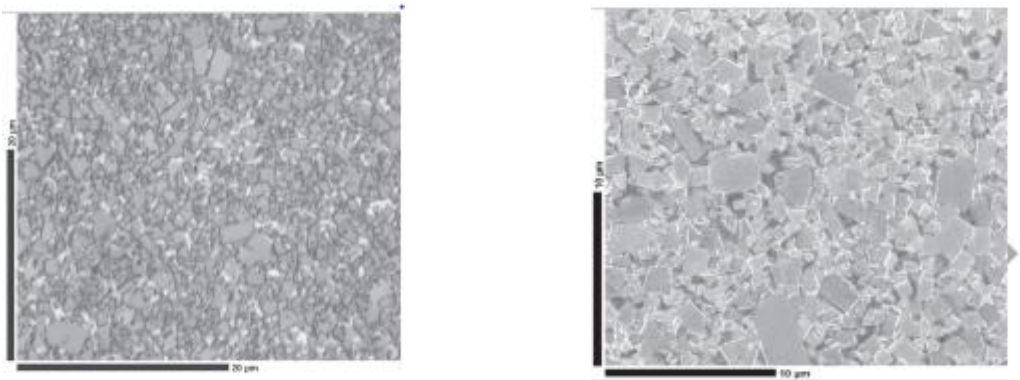


a) Nano — Optical b) Nano — SEM



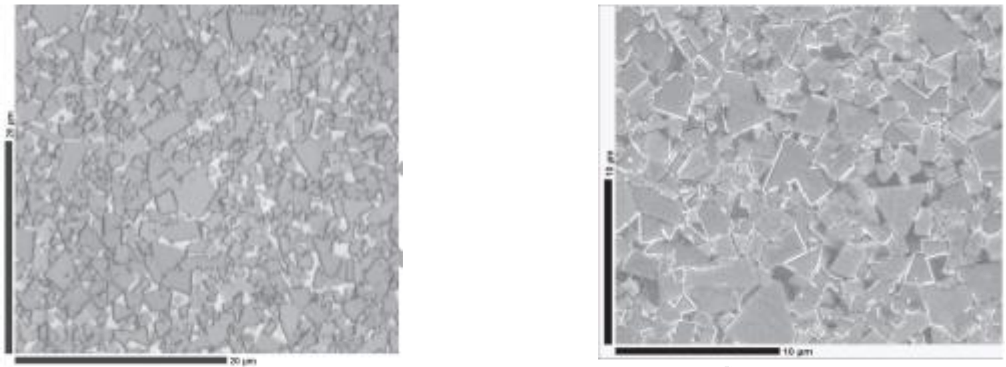
c) Ultrafine — Optical d) Ultrafine — SEM

FIG. 2 α-PHASE (WC) — OPTICAL AND SCANNING ELECTRON MICROSCOPE (SEM), NANO AND ULTRAFINE



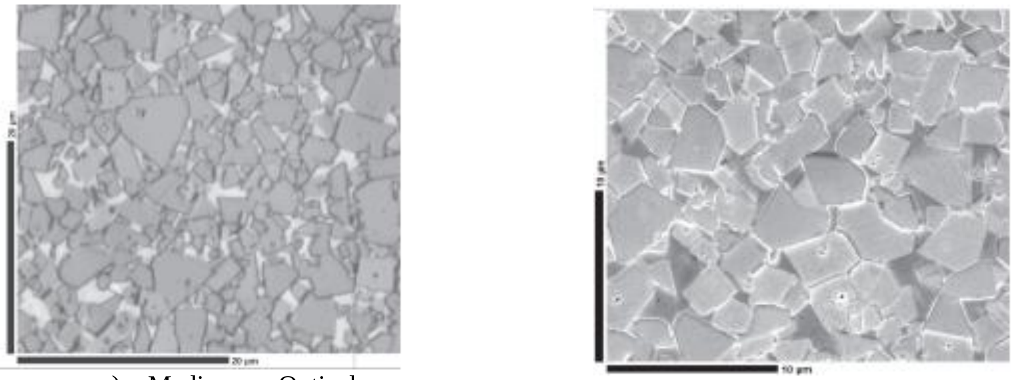
a) Submicron — Optical

b) Submicron — SEM



c) Fine — Optical

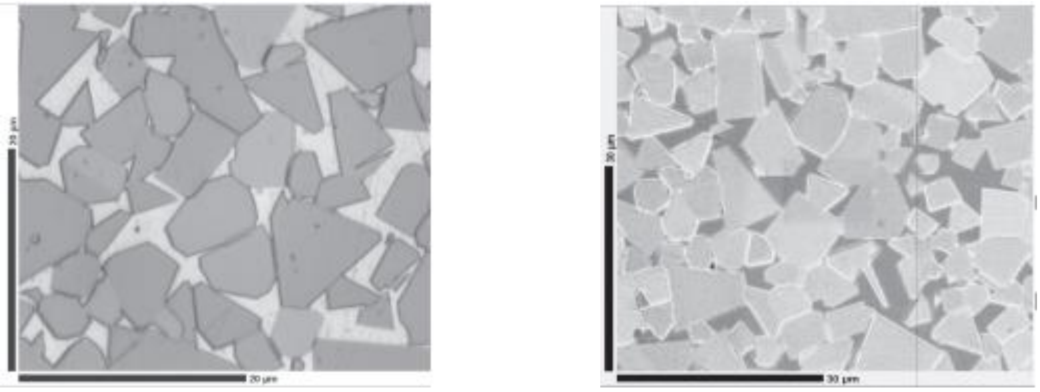
d) Fine — SEM



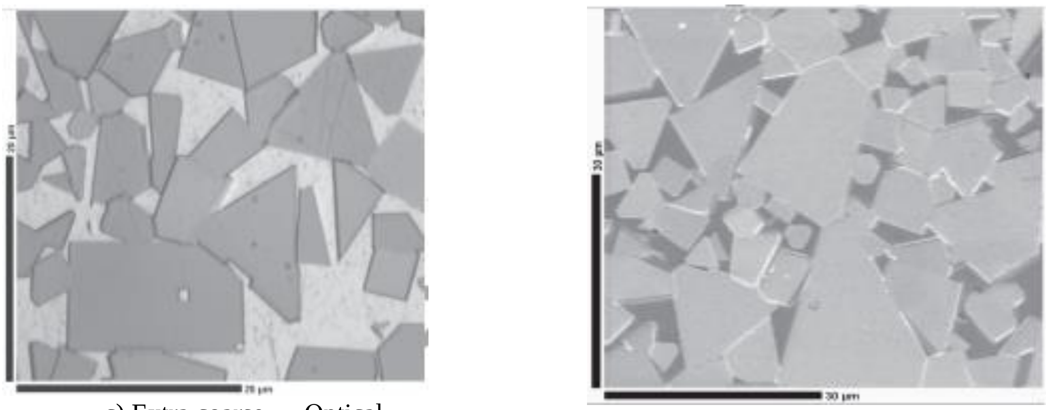
e) Medium — Optical

f) Medium — SEM

FIG. 3 α-PHASE (WC) — OPTICAL AND SCANNING ELECTRON MICROSCOPE (SEM), SUBMICRON, FINE AND MEDIUM



a) Coarse — Optical
b) Coarse — SEM



c) Extra coarse — Optical
d) Extra coarse — SEM

FIG. 4 α-PHASE (WC) — OPTICAL AND SCANNING ELECTRON MICROSCOPE (SEM), COARSE AND EXTRA COARSE

ANNEX A

(*Foreword*)

COMMITTEE COMPOSITION

Powder Metallurgical Materials and Products Sectional Committee, MTD 25

Organization	*Representative(s)*
Indian Institute of Technology Kanpur, Kanpur	Dr Anish Upadhyay **(*Chairperson*)**
Bhabha Atomic Research Centre, Mumbai	Prof Amit Sinha
Bharat Heavy Electrical Limited, New Delhi	Shri Vivek Arya Shri Bharat Kumar Pant (*Alternate*)
Controllerate of Quality Assurance, Ichapur	Shri A. Mitra Shri T. K. Prusty (*Alternate*)
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CSIR − National Metallurgical Laboratory, Jamshedpur	Dr V. C. Srivastava
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Member Secretary
Shri G. Ram Sai Kumar
Scientist 'B'/Assistant Director
(Metallurgical Engineering), BIS

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BUREAU OF INDIAN STANDARDS

Headquarters:

Manak Bhavan, 9 Bahadur Shah Zafar Marg, New Delhi 110002
Telephones: 2323 0131, 2323 3375, 2323 9402 *Website*: www.bis.gov.in

Regional Offices: *Telephones*

Central : 601/A, Konnectus Tower -1, 6th Floor, DMRC Building, Bhavbhuti Marg, New Delhi 110002 — 2323 7617

Eastern : 8th Floor, Plot No 7/7 & 7/8, CP Block, Sector V, Salt Lake, Kolkata, West Bengal 700091 — 2367 0012, 2320 9474

Northern : Plot No. 4-A, Sector 27-B, Madhya Marg, Chandigarh 160019 — 265 9930

Southern : C.I.T. Campus, IV Cross Road, Taramani, Chennai 600113 — 2254 1442, 2254 1216

Western : Plot No. E-9, Road No.-8, MIDC, Andheri (East), Mumbai 400093 — 2821 8093

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